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METHOD FOR DETERMINATION OF
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INDIAN STANDARDS INSTITUTION

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Indian Standard

METHOD FOR DETERMINATION OF SOLUBILITY OF WOOL IN ALKALI

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Indian Standard

METHOD FOR DETERMINATION OF SOLUBILITY OF WOOL IN ALKALI

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 4 February 1966, after the draft finalized by the Textile Chemistry Sectional Committee had been approved by the Textile Division Council.

0.2 The solubility of wool in alkali provides a useful index of the extent of the change in its chemical properties brought about by certain agencies. Wool, when treated with acids, oxidizing or reducing agents, or when exposed to heat or light, increases in solubility, whereas when treated with mild alkali, which is normally used in processing, or when treated with cross-linking agents, decreases in solubility. The solubility of wool depends on the severity of the treatment. This method of test is most useful when an un-treated control sample is available and when the nature of treatment of the sample under test is known. This method is useful as a control in processing. When the sample has been treated by two different agencies having opposite effects on the solubility, the interpretation of the results, even when an untreated controlled sample is available, is difficult and other tests are necessary to supplement the information.

0.3 Considerable assistance has been taken from B.S. 3568 : 1962 'Method of Test for the solubility of wool in alkali' issued by the British Standards Institution.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes a method for determining the solubility of wool in alkali applicable to all-wool textiles in any form such as fibre, yarn and fabrics.

2. PRINCIPLE

2.1 A sample under test is immersed in 0.1 N sodium hydroxide solution under specified conditions of time, temperature and volume. The loss in

*Rules for rounding off numerical values (*revised*).

weight is determined as the difference between the dry weights of the sample before and after treatment.

3. SAMPLING

3.1 Lot — The quantity of fibre, yarn or fabrics from the same batch of processing shall constitute a lot.

3.2 Sampling shall be carried out according to the relevant procedure given in Appendix A.

4. ATMOSPHERIC CONDITIONS FOR TESTING

4.1 The test shall be conducted in the prevailing room conditions.

NOTE — Since dry weights are determined, it is not necessary to condition the sample.

5. APPARATUS

5.1 Water-Bath — capable of maintaining a temperature of $66^{\circ} \pm 0.5^{\circ}\text{C}$.

5.2 Stoppered Conical Flasks — of 250 ml capacity.

5.3 Sintered-Glass Filtering Crucibles — of 30 ml capacity with a pore size of 90 to 150 microns and provided with ground-glass stoppers. If stopper is not available, the crucible should be covered with a watch-glass during cooling and weighing.

6. REAGENTS

6.0 Quality of Reagents — Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS : 1070-1960*) shall be used wherever the use of water as a reagent is intended.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the experimental results.

6.1 Sodium Hydroxide Solution — 0.1 N.

6.2 Acetic Acid Solution — prepared by dissolving 10 ml of glacial acetic acid in sufficient amount of water and made up to 1 litre.

6.3 Light Petroleum — boiling range between 40° and 60°C .

*Specification for water, distilled quality (*revised*).

7. PREPARATION OF TEST SPECIMENS

7.1 From the test sample, take a representative sample (about 10 g) sufficient to provide fat and burr free wool for the following test specimens:

- a) One test specimen weighing 1.0 g for determining the dry weight (see 8.1);
- b) Two test specimens, each weighing 1.0 g, for the solubility test (see 8.2.1 and 8.3); and
- c) Two test specimens each weighing 2.0 g for determining the acid content (see B-2.1).

NOTE — The representative sample should be disintegrated and cut into shorter lengths of one centimetre. The sample should be brought to room temperature.

7.2 Extract the representative sample in a Soxhlet apparatus with light petroleum for one hour at a minimum rate of six extractions per hour. Allow the petroleum to evaporate and remove the vegetable and other foreign matter by hand picking.

8. PROCEDURE

8.1 From the extracted representative sample, take one test specimen weighing 1.0 g. Dry it in a weighing bottle at $105^{\circ} \pm 3^{\circ}\text{C}$ for three hours. Stopper the bottle and cool it in a desiccator and weigh. Remove the test specimen and weigh the weighing bottle and calculate the dry weight of the test specimen.

8.2 Pour 100 ml of sodium hydroxide solution into a flask. Stopper it loosely and fix it in the water-bath by any suitable means, so that the level of the water outside the flask is at least 2 cm higher than the level of the solution inside.

NOTE — This procedure is essential for precise control of temperature.

8.2.1 When the temperature of the sodium hydroxide solution reaches $65^{\circ} \pm 0.5^{\circ}\text{C}$, introduce carefully one test specimen weighing 1.0 g into the flask. Replace the stopper tightly. Shake the flask gently to ensure complete wetting of the specimen and replace it in the water-bath. Again shake the flask gently after 15, 30 and 45 minutes, the time of shaking not to exceed 5 minutes. Continue the reaction for 60 minutes. Transfer the contents of the flask to a weighed filtering crucible, at the same time drain the crucible by suction. Wash the flask with distilled water and collect the washings in the filtering crucible. Wash the residue in the crucible six times with water, draining completely between each wash. Fill the crucible twice successively with acetic acid solution. Allow it to stand for one minute and drain the crucible by suction. Wash the residue six times with distilled water draining completely between each wash.

Dry the crucible and the contents at $105^{\circ} \pm 3^{\circ}\text{C}$ for three hours. Stopper the crucible or cover it with a watch glass. Cool it in a desiccator and weigh. Repeat the operations of drying and weighing until constant weight is obtained.

8.3 Repeat the procedure prescribed in **8.2** and **8.2.1** with one more test specimen weighing 1.0 g.

8.4 From the representative sample (see **7.1**) take about 1 g of test specimen. Extract it with cold water with a liquor to material ratio of 50 : 1 for half an hour shaking the flask occasionally and determine the pH of the extract. If the pH of the water extract is less than 4.0, determine the acid content by the method given in Appendix B.

9. CALCULATION

9.1 Calculate the alkali solubility of wool as the loss in weight of the test specimen, expressed as a percentage of its calculated dry, fat and acid free weight by the formula given in **9.1.1** or **9.1.2**.

9.1.1 If the sample does not contain acid (that is, if the pH is 4.0 or greater than 4.0) calculate the alkali solubility of each test specimen by the following formula:

$$S = \frac{W_1 - W_2}{W_1} \times 100$$

where

S = solubility in alkali, percent;

W_1 = dry weight of the test specimen (see **8.1**); and

W_2 = dry weight of the residue (see **8.2.1**).

9.1.1.1 Calculate the average of the two results obtained as in **9.1.1**.

9.1.2 If the sample contains acid (that is, if pH is less than 4.0) calculate the alkali solubility of each test specimen by the following formula:

$$S = \frac{100 \left(100 \frac{W_1 - W_2}{W_1} - a \right)}{100 - a}$$

where

S = solubility in alkali, percent;

W_1 = dry weight of the test specimen (see **8.1**);

W_2 = dry weight of the residue (see **8.2.1**); and

a = percentage of acid (see **B-3.2**).

9.1.2.1 Calculate the average of the two results obtained as in **9.1.2**.

10. REPORT

10.1 Report the value obtained as in **9.1.1.1** and **9.1.2.1** as the alkali solubility of the sample.

APPENDIX A

(Clause 3.2)

PROCEDURE FOR SAMPLING

A-1. SAMPLING FOR FIBRE AND YARN

A-1.1 The lot of fibre or yarn shall be divided into the sub-lots, each weighing 200 kg or less.

A-1.2 Each sub-lot shall be tested separately.

A-1.3 From a sub-lot, 15 increments each approximately weighing 10 g will be taken from different parts so that a representative sample is obtained. All the increments thus collected shall be thoroughly mixed. This shall constitute a test sample.

A-2. SAMPLING FOR FABRICS

A-2.1 The number of pieces to be selected shall be in accordance with col 1 and 2 of Table 1. The pieces thus selected shall constitute the gross sample.

TABLE 1 NUMBER OF PIECES TO BE SELECTED AT RANDOM FROM THE LOT

NUMBER OF PIECES IN THE LOT	NUMBER OF PIECES TO BE SELECTED
(1)	(2)
Up to 100	3
101 „ 300	4
301 „ 500	5
501 and above	7

A-2.2 From each piece in the gross sample, selected according to **A-2.1** cut about 25 g of fabric from at least two different parts. The fabric cut out shall then be disintegrated by cutting into short pieces of less than 1 cm square. This shall constitute a test sample.

APPENDIX B

(Clause 8.4)

METHOD FOR DETERMINING THE ACID CONTENT

B-1. REAGENTS

B-1.1 Pyridine Solution — prepared by dissolving 5 g of pyridine in one litre of distilled water.

B-1.2 Sodium Hydroxide Solution — 0.1 N.

NOTE — This solution should be standardized by titration with standard potassium hydrogen phthalate solution.

B-1.3 Phenolphthalein Indicator — prepared by dissolving 0.5 g of phenolphthalein in 95 ml of ethyl alcohol and 5 ml of distilled water.

B-2. PROCEDURE

B-2.1 Place two test specimens each weighing 2 g in separate glass-stoppered conical flasks. Pour 100 ml of pyridine solution into each flask. Stopper the flasks and shake mechanically for one hour or allow the flasks to stand overnight after initial shaking to ensure complete wetting of the specimens. Decant the liquid from the wool, filtering through a plug of glass wool to retain fibrous material. Pipette out 50 ml of each filtrate in separate conical flasks. Add three drops of phenolphthalein indicator to each flask and titrate separately with 0.1 N sodium hydroxide till a faint or a light pink colour appears.

B-3. CALCULATION

B-3.1 Calculate the weight of acid as the percentage of the dry weight of each specimen by the following formula:

$$a = \frac{v \times k \times n}{W_1}$$

where

a = acid content, percent;

v = volume, in ml, of 0.1 N sodium hydroxide solution required to neutralize 50 ml of pyridine extract;

k = constant (see Note);

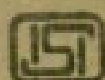
n = normality of the sodium hydroxide solution; and

W_1 = dry weight of 1 g specimen (see 8.1).

NOTE — The constant has the following values:

Acid	Value
Sulphuric acid	4.9
Formic acid	4.6
Acetic acid	6.0

B-3.2 Calculate the average of the values obtained as in B-3.1.



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